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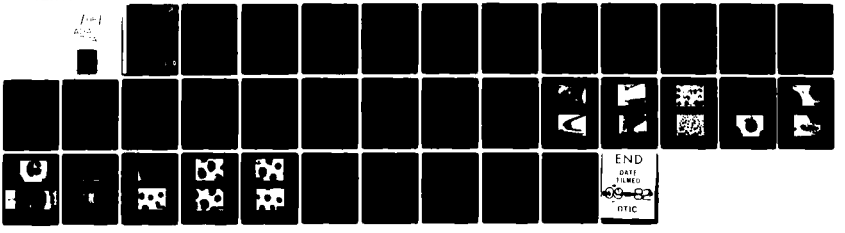

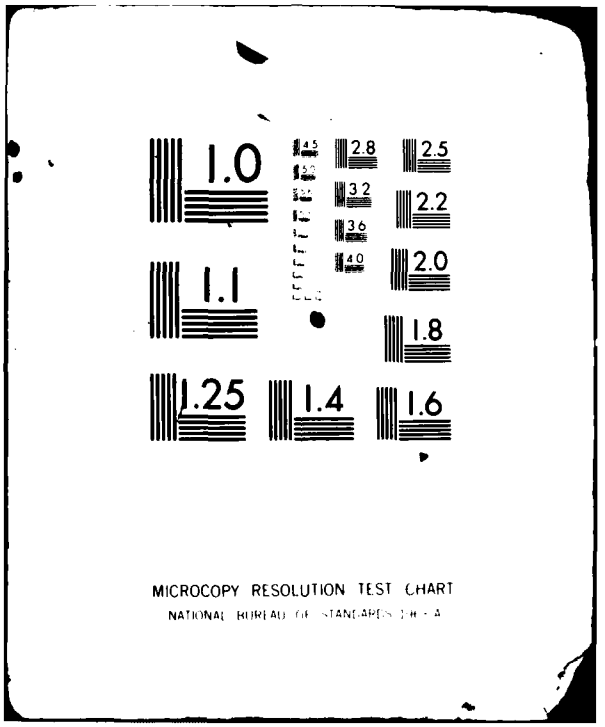
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AN INVESTIGATION OF INTERFACIAL REACTIONS IN METAL MATRIX COMPOSITES

March 1982

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ABSTRACT

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An Investigation of Interfacial Reactions in Metal Matrix Composites

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Abstract

The interfacial reaction between fibre and matrix materials was studied in G/Aℓ and FP/Mg composite systems. An intermediate layer of reaction product was necessary to prevent debonding in G/Aℓ composites during cooling from the fabrication temperature. The intermediate layer was identified as the spinel $MgAℓ_2O_4$ in the FP/Mg composite and the effect of this intermediate layer was to provide good bonding and load transfer from matrix to fibre and also to form bridges (necks) cross-linking the fibres. The spinel also acted as a diffusion barrier to reduce the role of fibre degradation by interfacial reaction at service temperatures.

1. Introduction

The advantageous employment of metal matrix fibrous composites as modern structural materials applications is dependent on the combination of low density and high stiffness which can be achieved consistently in the production and fabrication of such continuous fibrous composites into the shapes and sizes specified for these applications.

Composite materials which are candidates for future utilization are magnesium and magnesium alloys reinforced with graphite, boron, or such dispersed phases as FP ($A\ell_2O_3$) and SiC. There is also the possibility that aluminum/boron composites might be suitable for light weight, high strength and stiffness applications. The graphite and boron have very low densities so that the composites fabricated with either of these dispersed phases would have lower specific weights than composites of the same matrix material fabricated with such higher density dispersed phases as FP or SiC. The low density of magnesium and magnesium alloys render these metals and alloys exceptionally well suited for these applications.

All of the factors tend to indicate that the above composite materials would all be very well suited for light weight, high stiffness structural applications. Before that decision can be established, some potential problem areas must be considered. The present research program is intended to examine in detail the interfacial reactions in metal matrix composites.

2. Scope of Research

The fabrication of composite materials using matrices of metals and alloys and containing dispersed fibres (either oriented or randomly dispersed) or needles (whiskers) requires the use of elevated temperatures during the production of the basic materials or during the application or stiffening elements of composite to ordinary non-composite engineering

alloys already fabricated. This heating during fabrication or application of the composite or the heat treatment of the composite to achieve the optimum properties if a heat-treatable matrix alloy is used can result in interfacial reactions between the matrix and the dispersed phase, between any intermediates and either the matrix or the dispersed phase. These interfacial reactions result in the formation of reaction products and the depletion of either matrix or dispersed phase or intermediate. In this way, the resultant properties of the composite may depend greatly on the nature and extent of the reactions which have occurred at the interface throughout the composite material. In addition, the coefficients of thermal expansion of the matrix and of the dispersed phase are quite dissimilar in most of the systems being contemplated for helicopter applications. This means that composites fabricated at elevated temperatures will either have built-in stresses (residual stresses) or internal cracking or delaminations due to this disparity of coefficient of thermal expansion. The application of composites to an engineering structure by such methods as hot pressing also will result in the same types of reactions and thermal stresses or internal cracking or delaminations. The transverse properties of oriented fibre reinforced composites are often severely limited by these phenomena.

The present research effort was to have been devoted to studying interfacial reactions in FP ($A\ell_2O_3$)/magnesium and SiC/aluminum. The objective of this research was to provide new understanding of fibre/matrix interfacial reactions in these composites. This new understanding would then provide a basis for optimizing the properties of composite systems for use in Army helicopter drive systems and bridging through control and minimization of any damaging fibre/matrix interfacial reactions.

To investigate and understand the interfacial reactions which may occur during fabrication and use of the FP/Mg and SiC/Aℓ composite systems, continuous fibres in well-characterized metal matrices were used.

The as-fabricated composites were to be examined using such techniques as optical metallography, scanning electron microscopy, electron microprobe analysis, electron fractography, x-ray diffraction, etc. This would establish the microstructure and distribution of the constituents of the composite

and any products of interfacial reactions which had occurred during fabrication.

After fabrication, the composites were to be heated to service temperatures for specific times and evaluated as to microstructure and interfacial reaction products.

The strength and stiffness of as-fabricated and post-fabrication heat treated composites were to have been measured and the anisotropy determined.

Then, using the data as generated, the interfacial reactions would be controlled to provide those products at the interface which improved properties.

3. Limitations on Research Scope

The original contract called for two composite systems to be studied during the contract year, i.e., the FP/Mg and the SiC/Aℓ systems. Orders for the FP/Mg composite material were placed with DuPont and a two-three week shipment was offered. The material was delayed due to a backlog in the DuPont production schedule and, despite the efforts of the AMMRC to increase the priority attached to this order, the material was not received until five months after the date of the order.

The source of the SiC/Aℓ was to have been AVCO (Lowell).

Production problems at AVCO were encountered and, despite further efforts of both Brown University personnel and AMMRC, no SiC/Aℓ has been provided to this date.

Efforts to obtain SiC/Aℓ from other personnel and research groups at AMMRC were made, but no material was found to be available.

The initial delay in obtaining both composite materials prompted an initial investigation into interfacial reactions in some G/Aℓ provided by AMMRC. This work was then superceded at the end of December 1980 when the FP/Mg from DuPont was received and work was immediately commenced on that material. The unavailability of SiC/Aℓ resulted in the inability of the

present research investigation to look into the interfacial reactions in that composite system and the delay in determining availability prevented any substitute composite from being included in the program because of expected delays in obtaining any substitute material system.

The result of the supply problems and delivery problems was that the scope of the present research effort was limited to studying the interfacial reactions which had occurred during fabrication and during post-fabrication heat treatment in the FP/Mg system with some examination of the interfacial reactions which had occurred during fabrication in the G/Aℓ system. No work was performed on the SiC/Aℓ system and no time was available for mechanical strength and stiffness testing of the FP/Mg composite material.

During the initial period when receipt of the composites was anticipated, literature review was accomplished and calculations were made of the effect of the mismatch of coefficients of thermal expansion between the matrix and dispersed phase on the generation of residual stresses, the onset of yielding and debonding in generalized composites to help explain the effects of fabrication and post-fabrication heating in the real systems and also the anisotropy of strength in real systems.

In addition, during the waiting period, theoretical calculations were made of the interfacial reactions which would, most probably, be encountered in the systems to be studied in this research program.

4. Review of Literature

The interface of a fibre reinforced metal matrix composite material is defined as the area of separation between fibre and the metal matrix. It is known that most failures of metal matrix composites had been attributed to weakness at the fibre/matrix interface. Many times debonding occurs at the interface when the composite is stressed well below its theoretical (ROM) strength. This phenomenon occurs most frequently in non-metal, ceramic fibre/metal matrix composite systems. In theory, the interface must satisfy the following conditions:

1. **There must be thermodynamic wetting between the fibre and matrix.**

2. The bonding forces between matrix and the fibre must act over an area large enough to insure load transfer from matrix to fibre.

3. Bonds formed must be strong and stable for long times at service temperatures.

4. Any reaction zone formed at the interface between the fibre and the matrix must be small in thickness compared to the fibre diameter.

5. The coefficient of thermal expansion of the metal matrix and the fibre materials must be sufficiently similar that thermal stresses will not be great enough to destroy or weaken the bond at the interface.

Even though much is known about why and how most unsuccessful fibre reinforced metal matrix composites have failed, little is known about how to prevent it. For example, most ceramic materials are not wetted by metals and often a non-uniform or porous interface is formed which results in interfacial failures. Ceramics that are wetted by metals often form a brittle intermetallic compound at the interface which has proven to be detrimental to mechanical properties. The coefficient of thermal expansion of ceramics is usually lower than that of metals, and residual stresses at the fibre/matrix interface results. This can cause yielding and debonding after fabrication and failure during service conditions.

One of the early successful fibre reinforced metal matrix composites was silicon carbide-coated boron fibre reinforced aluminum matrix composites. Observations of composite fracture surfaces had indicated the considerable strength of the fibre-matrix interface and had shown that interfacial failure was seldom a mode of composite fracture. Prewo and McCarthy (1) stated that the reasons for the high interfacial strength was related to the intimate contact established between matrix and fibre during composite fabrication. This interfacial bond was established without the generation of observable reaction products. It was also shown that the boron fibre was structureless while the silicon carbide, deposited as a diffusion barrier layer, was made up of many elongated crystalline regions whose primary axes were perpendicular to the boron-silicon carbide interface. The surface structure of this coating was related to the growth pattern

of the silicon carbide and provided a serrated surface which was replicated to the finest observable detail by the aluminum matrix.

More recently successful composites were made of graphite or polycrystalline alumina (FP) fibres in aluminum. These materials were fabricated by the Sodium Process. Composites with full theoretical strength were prepared by means of this process if rayon-base graphite, PanI graphite, or FP fibres were used. The sodium process for the preparation of fibre-reinforced aluminum composites consisted first of wetting the fibres with molten sodium, then forming protective intermetallic compound fibre coatings by one or more other molten metals, and finally by displacing all metal except the fibre coating with a final bath of molten aluminum or aluminum alloy. Goddard (2) found that liquid sodium penetrated graphite by intergranular diffusion or by intercalation in which such compounds as $C_{64}Na$ may have formed. Sodium acted as a protective and wettable coating on the fibres. The second bath usually consisted of molten tin, its major function being to dissolve and displace the sodium. The tin was miscible with both sodium and aluminum, whereas the sodium was totally immiscible with aluminum. Since sodium and tin form a series of intermetallic compounds, less than 1.5% magnesium was added to either the tin or sodium bath. This resulted in the formation of Mg_2Sn which acted to stabilize the sodium-rich fibre coating. The final aluminum bath dissolved and displaced the tin, which was possible because of the complete miscibility of these metals. The metallurgical reactions that occurred at the fibre-matrix interface during this process provide insight into potential methods of forming other protective fibre coatings suitable for other fibre-matrix composite systems.

In 1978, a new and successful composite consisting of Alumina (FP) fibre in magnesium or aluminum was introduced by DuPont(3,4). These composites were fabricated without any intermediate wetting agent. Tensile tests of these composite materials up to fracture indicated that the interfaces produced were strong enough to permit the transfer of loads at strengths in the order of 250 to 350 MPa. Levi, Abbaschian and Mehrabian (5) studied interfacial reactions during fabrication of FP/aluminum alloy and observed that interactions in the Aℓ-Cu system produced a distinctive accumulation of

copper around the fibres in the form of discrete particles of a Cu-rich phase which disappeared with heat treatment. It was suggested that $CuAl_2O_4$ may have been present along with α-Al_2O_3 and possibly $CuAl_2$ in the interaction zone. Interactions between the Al_2O_3 fibres and the Al-Mg alloy matrix resulted in the formation of a Mg-rich region around the fibres which was retained during heat treatment. This was attributed to the presence of $MgAl_2O_4$ and MgO at the fibre boundary in addition to α-Al_2O_3. Changes in appearance of the interaction zone were also observed for different magnesium contents in the alloy. Additions of small amounts of magnesium to the Al-Cu alloy significantly reduced the extent of the interaction observed. Both magnesium and copper enrichments around the fibre were detected in this stage, and these zones of enrichment were still present after heat treatment. Experimental observations indicated that $MgAl_2O_4$, α-Al_2O_3 and possibly $CuAl_2O_4$ coexisted in the interfacial zone. The results of this study (5) suggested that a compound of the aluminate type may form on the fibre surface and provide the required bond with the matrix. This reaction would be enhanced in the presence of oxygen during the fabrication step.

From these previous studies it can be seen that an interface formed by intimate contact between the fibre and metal matrix, an infinitely thin wettable intermediate layer, or simply a thin stable intermetallic layer, can produce a strong interface and high composite strength.

5. Experimental Procedure

The general procedure for examining the various specimens of composite materials consisted of:

1. Sectioning - cutting the samples out of the bulk as-received material in the proper shape and orientation so that they could be studied in detail by the procedures detailed below.
2. Mounting - the sections cut from the bulk material were mounted in epoxy cold-mounting material so that they could be prepared metallographically.
3. Metallographic preparation - the as-mounted samples were carried through various grits of polishing material from the 320 and 600 silicon carbide papers to 6 micron, 1 micron, 0.25 micron diamond papers (prepared in the

laboratory), and then to standard diamond polishing wheels of 6 micron and 0.25 micron sizes.

4. Optical examination using standard metallographic microscopes to observe the uniformity of the dispersion of the fibres and fibre bundles in the matrix and other unusual features which might influence the properties of the composite.
5. Sputtering of conductive material (gold) onto the surface of the mount but not onto the polished surface of the composite specimen in the mount.
6. Scanning Electron Microscopy using an AMR 1000A scanning electron microscope in the secondary electron mode.
7. Electron microprobe analysis of the composite specimens using EDAX analysis (Energy Dispersive) in the AMR 1000A and wave length dispersive analysis (in some cases) using an ARL - SEM/EMX to determine concentration gradients across the fibre/matrix interfaces. (All probe analyses were performed on samples coated with carbon and not gold.)
8. X-ray diffraction of bulk as-received samples of composite materials to determine the phases and compounds present in the composites.
9. X-ray diffraction of powdered composites (ground) and prepared with an internal reference standard for determination of the concentration of interfacial reaction products in the composite.

The procedures described above were utilized for examination of the composite materials in their as-received (i.e., as-fabricated) conditions and again after they had been subjected to heating for various lengths of time at a selected temperature. Since many of the above procedural steps are standard within metallurgical laboratories, there is no need to discuss them in detail here. Those procedures which are not standard will be discussed in detail.

The sectioning of the FP/Mg required special procedures before it could be accomplished with no damage to the specimens. The very hard nature of

the alumina (FP) fibres coupled with the soft and low melting point magnesium matrix caused such heating when cutting with the usual equipment at room temperature or when cutting with water cooled diamond cut off wheels that the samples became hot and deformed while the cutting surfaces became loaded with magnesium and reduced the cutting efficiency.

After attempting various techniques, a simple hacksaw was found to be effective when the specimen was held in a vise and the entire vise and hacksaw were maintained in an ice-filled bath. This allowed sections to be cut by hand with no appreciable heating or plastic deformation to the specimens.

The use of diamond paste in metallographic preparation and the use of higher than normal pressures and high speeds on the diamond polishing wheels was because of the difference between the hardness of the dispersed FP and the magnesium matrix. By using diamond wheels at high speed and with higher than normal pressures, it was possible to prepare metallographic samples which did not have the magnesium removed at a greater rate than the alumina and resultant height changes on the plane of polish.

The usual method of preparation of non-conductive specimens for scanning electron microscopy is to coat them with gold or carbon. In the instant case, the mounted and polished specimens were comprised of a conductive matrix but a non-conductive aluminum oxide and a non-conductive epoxy mount. The small size of the fibres prevented their non-conducting nature from causing charge build up, but the surface of the mount was large in area relative to the specimen within the mount and that non-conducting area had to be made conductive. To do this, a tape was placed over the mount and the specimen within the mount. The tape was then cut with a surgical scalpel so that the tape could be peeled away from the epoxy surface while the tape still protected the specimen surface. This sample was then sputtered with gold. The tape was then removed from the specimen surface and the mount placed on the stage of the scanning electron microscope and examined.

Once the scanning electron microscopy had been performed, it was necessary to coat the specimen surface with carbon using an evaporator before microprobe analysis could be performed. The specimen was polished

so that the gold was removed from the epoxy surface and then the carbon layer was deposited over the mount and specimen areas. The sample was then returned to the AMR 1000A for the energy dispersive analysis and determination of concentration gradients or to the ARL-SEM/EMX for the wave length dispersive analysis.

For the quantitative determination of the concentration of interfacial reaction product present in the sample, it was necessary to employ powder x-ray diffraction methods. The composite was first taken to whatever stage of treatment, i.e., as-fabricated or heat treated post fabrication, and then ground using a mortar and pestle. An internal standard of silicon powder was then added in measured quantity to the specimen of ground composite. This sample was then made into an acetone glycolphthalate slurry and deposited onto a glass microscope slide for x-ray diffraction analysis. The peak areas of heat treated samples are compared with those of non-heat treated samples and the comparison of the areas of the silicon peaks in both samples allows a quantitative determination to be made of the reaction product concentration.

To determine the effect of post-fabrication heat treatment on the nature and extent of the interfacial reaction, the FP/Mg composite material was heated at a temperature of 350^{o}C (to simulate the temperature of the helicopter transmission) for various lengths of time ranging from 1 hour to 500 hours. The samples were cut from the as-fabricated material and heated in air for the required length of time and then cooled to room temperature. Once heat treated, the samples were subjected to the examination procedures detailed above.

6. Results

(a) Residual Stress Calculations

The mismatch between the coefficients of thermal expansion of the dispersed phase and the matrix in a composite material are such that when a bond is achieved between the two constituents at the fabrication temperature, cooling to room temperature can result in the generation of residual stresses, yielding of the matrix and possible debonding between the matrix and fibres.

Using the mathematical models and formulas developed by Burgreen (6), various residual stresses which develop on cooling a composite with a 50 v/o dispersed phase over a temperature interval ΔT, are given by the relation

$$P = \frac{(\alpha_1 - \alpha_2)(\Delta T)}{\frac{1}{E_2}\left(\frac{E_2}{2G_2} + 2\right) - \frac{1}{E_1}}$$

σ_{1r} = radial stress on fibre = -P

σ_{1t} = tangential stress on fibre = +P

σ_{2r} = radial stress on matrix = -P

σ_{2t} = tangential stress on matrix = 3P

where

1 refers to fibre

2 refers to matrix

E ≡ Elastic modulus

G ≡ Shear modulus

α ≡ coefficient of thermal expansion

ΔT ≡ Temperature change

(b) Thermodynamic Calculations

In considering the possible interfacial reactions which could possibly occur in the systems FP/Mg, the Gibbs Free Energies of formation of the various possible reaction products were considered for all possible compounds involving aluminum, magnesium and oxygen.

The compound with the largest negative Gibbs Free Energy of Formation at the fabrication temperatures was the compound $MgAl_2O_4$. This is a spinel structure and is similar in crystallography and chemistry to the spinel postulated by Richman, Levitt and DiCesare (7) as being the product of the interfacial reaction in the G/Mg system, that spinel being $MgTi_2O_4$.

The verification of the existence of the spinel, $MgAl_2O_4$, in the as-fabricated and heat treated post fabrication samples of FP/Mg composite was provided by x-ray diffraction and will be described below.

(c) G/Aℓ Composite System

Oblique sections of G/Aℓ were mounted and metallographically polished. Upon examination in the scanning electron microscope, it was determined that where there was good bonding between the graphite fibre and the matrix there was, indeed, an intermediate layer which was in good contact with the matrix and with the fibre. Where the fibres had debonded from the matrix, there was no intermediate layer between them.

A typical scanning electron micrograph of the intermediate layer between a graphite fibre and the aluminum matrix is shown in Figure 1 at 68,000X. While there is some gap just below the fibre in this micrograph, the bonding elsewhere is very good. A debonded fibre from the same sample is shown at 29,000X in Figure 2. This fibre has completely separated from the matrix and there is also no intermediate layer of interfacial reaction product serving to provide the bond to both the matrix and the fibre in this case.

To prove that the intermediate layer is different in chemical composition and in crystal structure from the matrix, a sample area was selected where there was an intermediate layer (see Figure 3). This sample was then etched with Kellers reagent and then re-examined in the SEM. A similar area of the specimen is shown in Figure 4 and the attack of the etchant on the matrix has resulted only in small pits being formed whereas in the intermediate layer, the etchant has severely attacked the interfacial reaction procuct.

(d) FP/Mg Composite

The DuPont FP/Mg composite was sectioned and polishing procedures were developed to reveal the microstructure and fine structure (in the SEM). Normal polishing methods employing either aluminum oxide abrasives result in the matrix being polished away while the fibres remain raised. A typical low power scanning electron micrograph of this is shown in Figure 5. Normal pressure and speed using diamond polishing wheels also results in leaving the fibres raised high above the polished-away matrix.

Using the special polishing procedures described above, it was possible to achieve a polish with the matrix and fibres being in the same plane. This is shown in the scanning electron micrograph of Figure 6. An optical micrograph of this same specimen is shown in Figure 7.

The optical micrograph (Figure 7) shows that the FP fibres are not completely uniform in their dispersion in the magnesium matrix. There are many cases where two fibres are actually in contact with one another (see Figure 5) and this contact can be seen in even greater detail in Figure 8 which shows the neck formed between two fibres and their interfacial reaction layers. This interface overlap is also shown in Figure 9.

The intermediate layer on one of the fibres in Figure 9 is relatively free of pores while the other intermediate layer is quite porous. This porosity of the interfacial reaction product is shown in more detail in Figure 10. A similar juxtaposition of a porous and non-porous intermediate layer pair is also visible in the micrograph of Figure 8.

In order to determine the concentration gradient from the fibre through the intermediate layer, it was possible to perform electron microbeam probe analysis (EDAX) on the specimens and to plot the concentration gradient. The exact positions on the surface of the specimen where the counts were taken is indicated on the micrographs by white dots.

In Figure 11 is shown a scanning electron micrograph of a fibre section with its intermediate layer and the adjoining matrix. The white dots represent spots at which the concentration gradient was measured quantitatively. In Figure 12, a set of concentration measurement points runs across the fibre and intermediate layer from matrix to matrix. The graph of magnesium and aluminum contents across this line of measuring points is presented in Figure 13.

This graph shows that there is a peak in the aluminum concentration within the intermediate layer and an abrupt drop-off of aluminum content in the matrix. The magnesium content is seen to rise slowly in the intermediate layer.

Another concentration gradient was plotted across two fibres, their intermediate layers, and the neck formed between those two intermediate layers. The positions of the measuring points are shown in the micrograph of Figure 14 and the actual profiles in Figure 15.

The samples of FP/Mg were then heat treated for various lengths of time at 350^{o}C and cooled to room temperature. The scanning electron

micrographs of specimens of composite after varying times at temperature are shown in Figures 16 through 20. While many areas were studied and micrographs were taken from each time, representative micrographs are presented here.

The as-fabricated composite is shown in the scanning electron micrograph of Figure 16. After one hour, the structure is shown in Figure 17, after three hours in Figure 18, after fifty hours in Figure 19, and after 100 hours in Figure 20.

The intermediate layer increases only slightly (as evidenced by examination and measurement of the micrographs) as a function of the heating time. For this reason x-ray diffraction methods were developed to show quantitatively the change in concentration of the interfacial reaction product. The initial x-ray diffraction runs indicate that there is, indeed, an increase of the interfacial reaction product as a function of time at temperature.

The nature of the intermediate layer is such that it acts to retard the diffusion of magnesium through the spinel crystal structure and thus prevent any further reduction of the fibre diameter (with consequent loss of strength of the composite). The intermediate also acts to form a bridge (by neck formation) between adjacent fibres in the matrix and, thus, establish a cross linking between the fibres.

7. Conclusions

(1) Interfacial reactions are necessary to promote bonding and wetting between fibres and matrix in G/Aℓ and FP/Mg composites.

(2) In G/Aℓ composites, where no intermediate layer is formed, debonding of the fibres occurs during cooling from the fabrication temperature. Where an intermediate layer is formed, bonding from fibre to intermediate layer to matrix resists debonding under residual stresses and allows load transfer on application of service conditions.

(3) Residual stresses, yielding and debonding can occur on cooling from the fabrication temperature to room temperature due to the mismatch of coefficients of thermal expansion. This is further enhanced by post-fabrication heat treatment or service at elevated temperatures.

(4) The interfacial reaction product in FP/Mg composites is the spinel $MgAl_2O_4$.

(5) The intermediate layer of $MgAl_2O_4$ acts as a diffusion barrier and retards further reaction and growth of this layer.

(6) The intermediate layer of $MgAl_2O_4$ forms bridges or necks between those fibres in close juxtaposition to one another, which bridging should provide for improved transverse strength.

(7) There is an increase in the thickness of the intermediate layer of interfacial reaction product as a function of time at temperature (post-fabrication) which can be determined by x-ray diffraction studies.

(8) The mechanical properties of FP/Mg composites are derived from the interfacial reaction product forming between fibres and matrix.

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Figure 1. Graphite Fibre in Aluminum Matrix with Intermediate Layer of Interfacial Reaction Product.

Figure 2. Graphite Fibre in Aluminum Matrix. No intermediate layer is present and fibre has debonded from matrix.

Figure 3. Intermediate Layer Between Graphite Fibre and Aluminum Matrix.

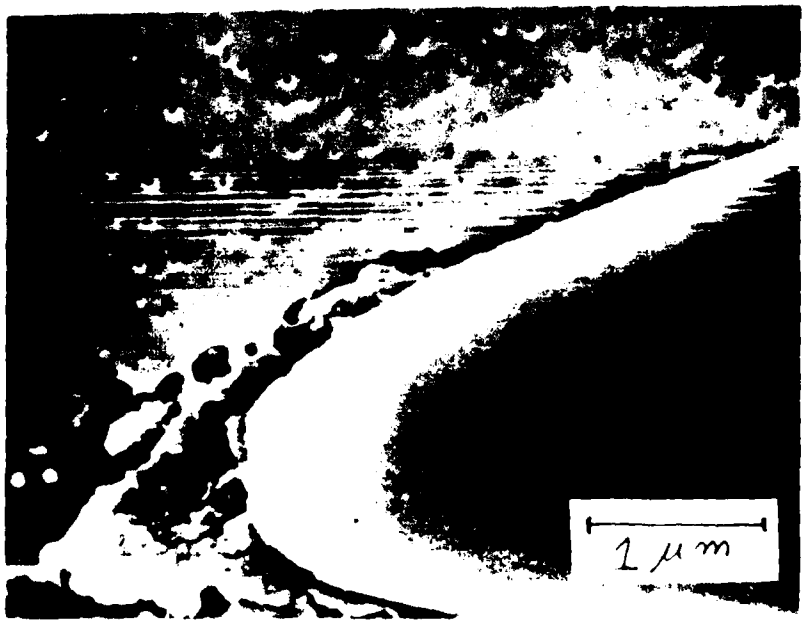


Figure 4. Effect of Kellers Etch on Aℓ Matrix and Intermediate Layer.

Figure 5. FP/Mg after Polishing with $A\ell_2O_3$.

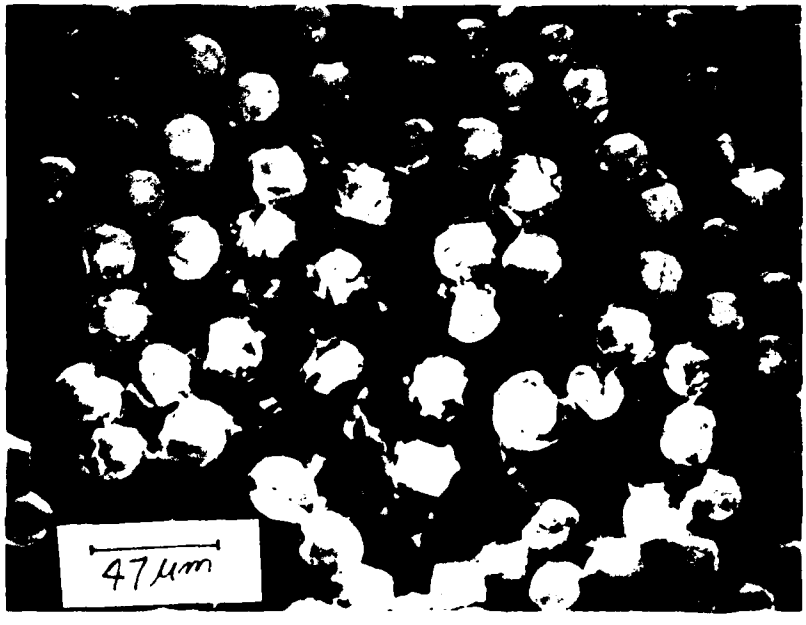


Figure 6. FP/Mg After Polishing with Diamond of High Speed and High Pressure.

Figure 7. Optical Micrograph of FP/Mg. Note dispersion of fibres.

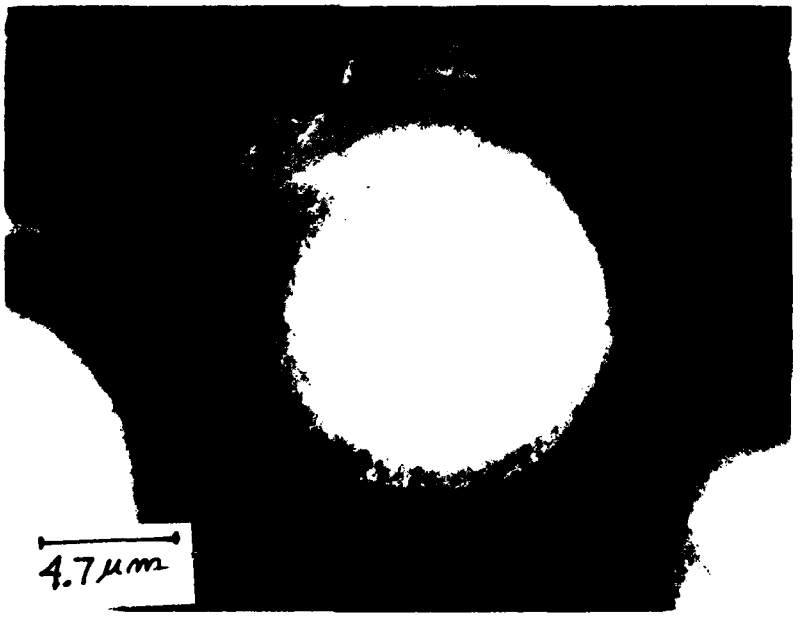


Figure 8. Interfacial Reaction Product Formed in FP/Mg During Fabrication.

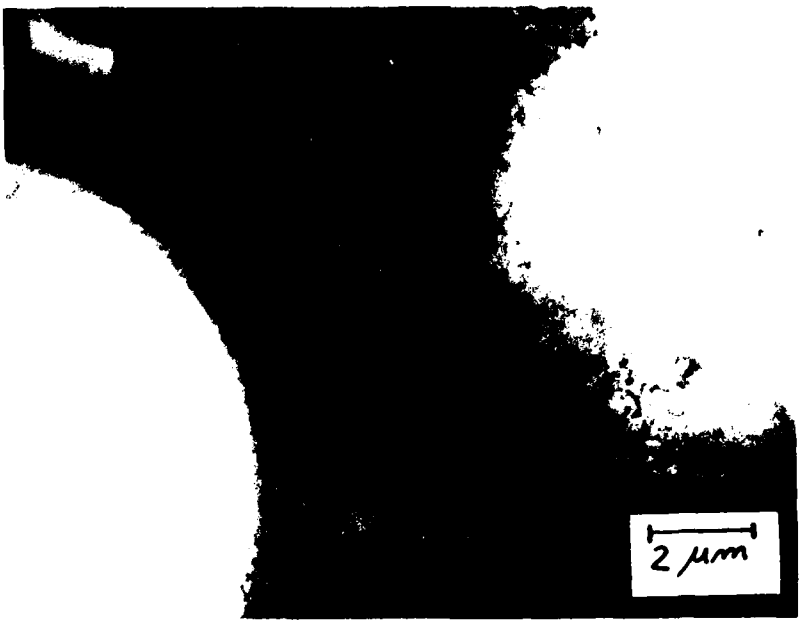


Figure 9. Intermediate Layer Bridging During Fabrication in FP/Mg.

Figure 10. Porous Nature of Interfacial Reaction Product in FP/Mg.

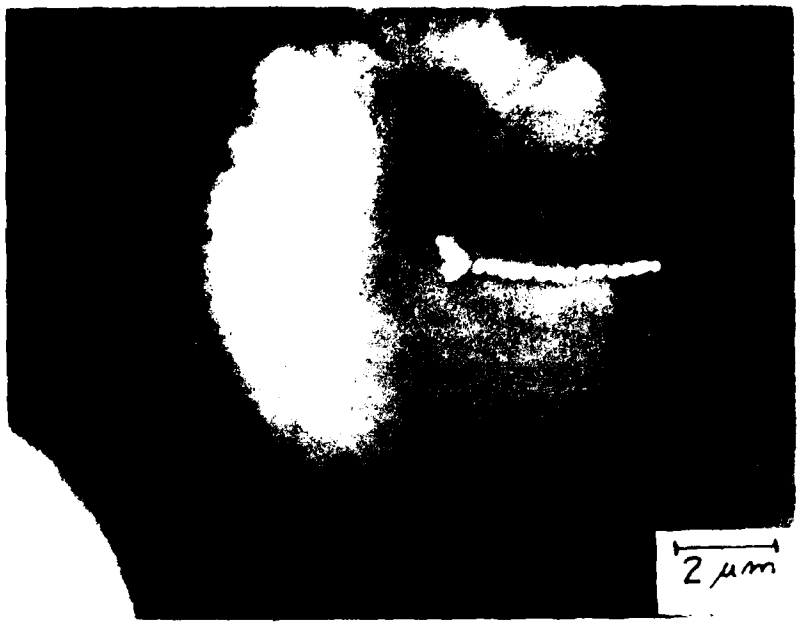
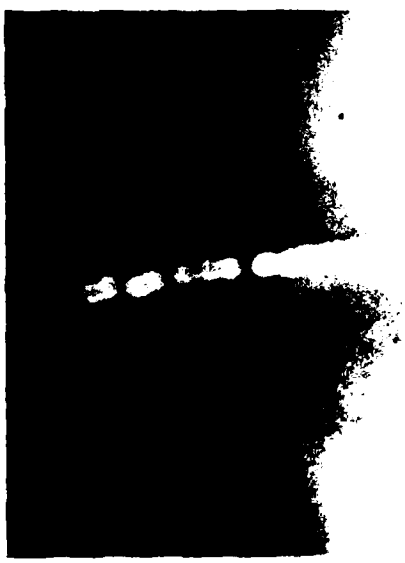


Figure 11. Microprobe Analysis Taken of Spots Shown on SEM of Fibre and Intermediate Layer in FP/Mg.



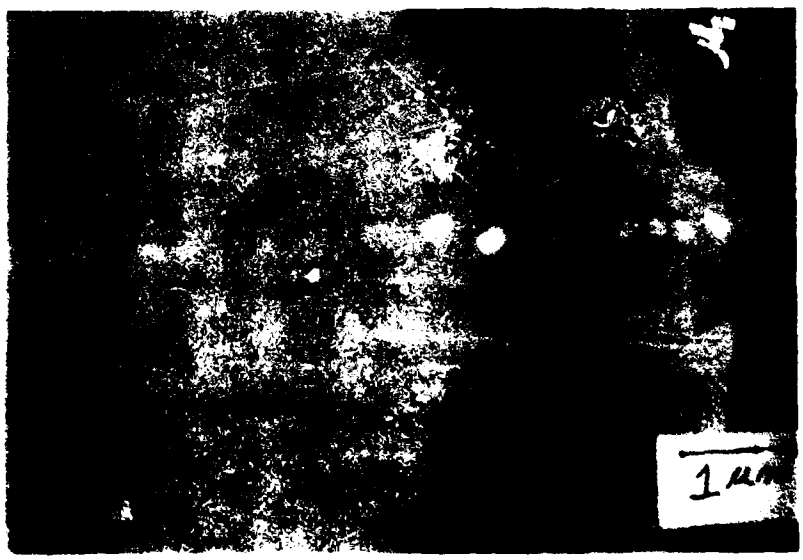


Figure 12. Another Set of Microprobe Spots in FP/Mg.

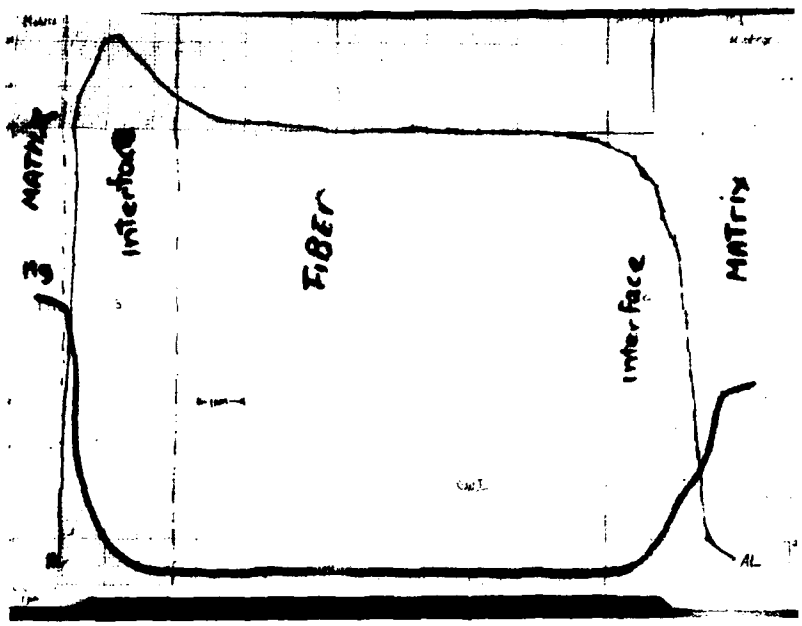


Figure 13. Concentration Gradients of Mg and Aℓ from Probe Trace of Figure 12.

Figure 14. Probe Trace Across Bridge Between Two Fibres of FP/Mg.

Figure 15. Concentration Gradients of Mg and Aℓ from Probe Trace of Figure 14.

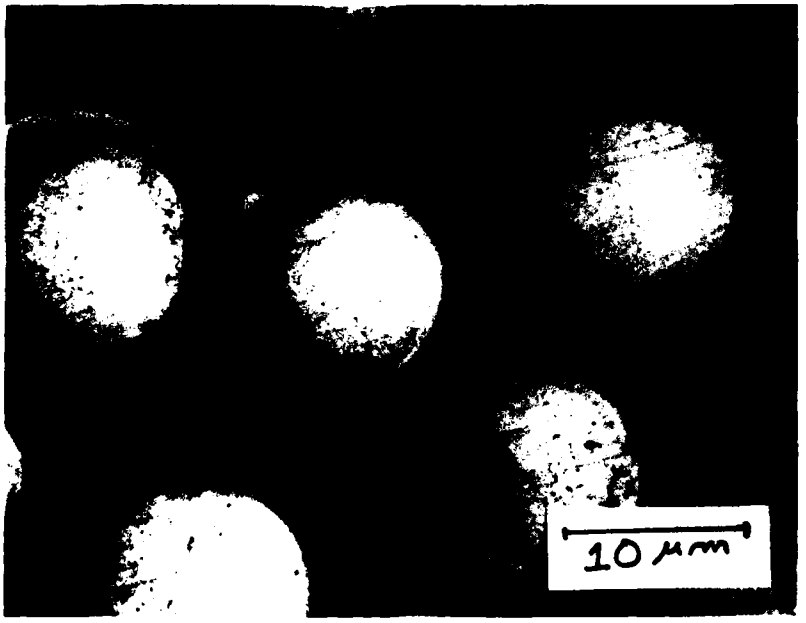


Figure 16. As-Fabricated FP/Mg.

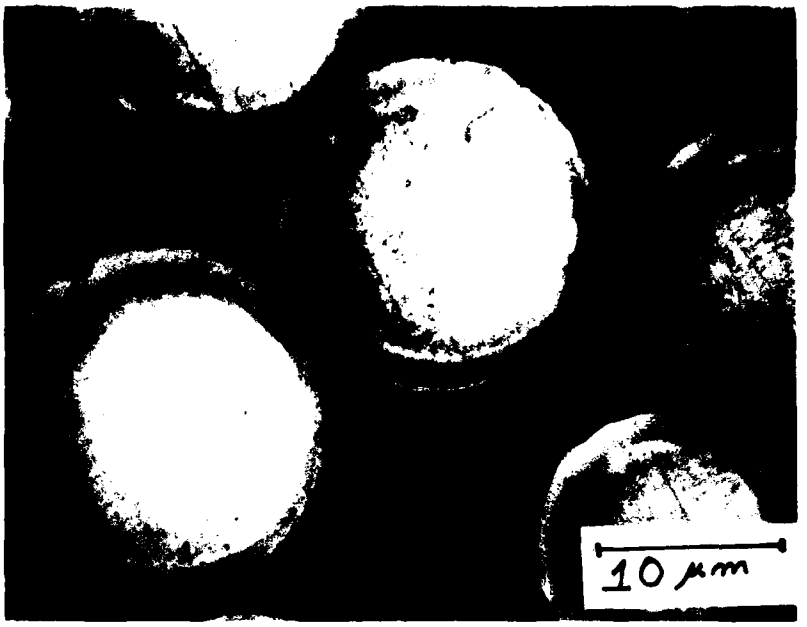


Figure 17. FP/Mg Heated for 1 hour at 350°C.

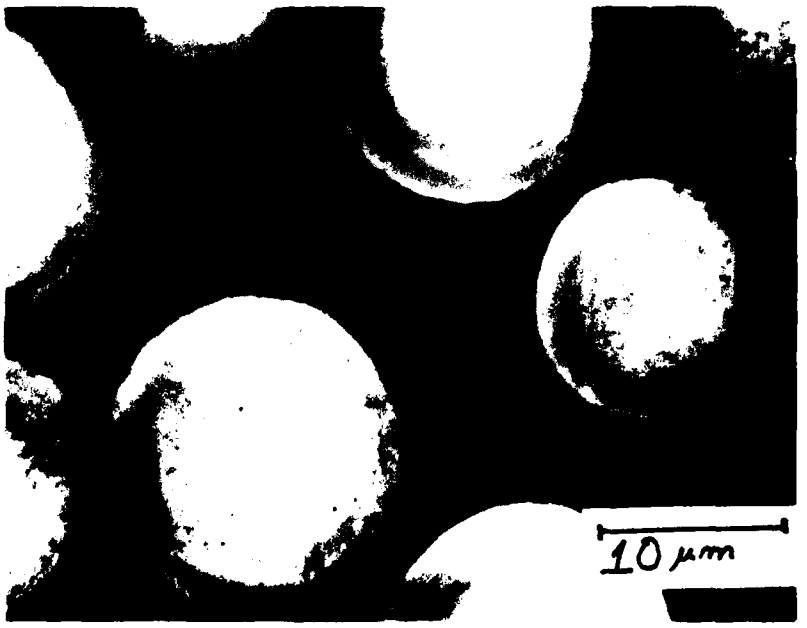


Figure 18. FP/Mg Heated for 3 hours at 350°C.

Figure 19. FP/Mg Heated for 50 hours at 350°C.

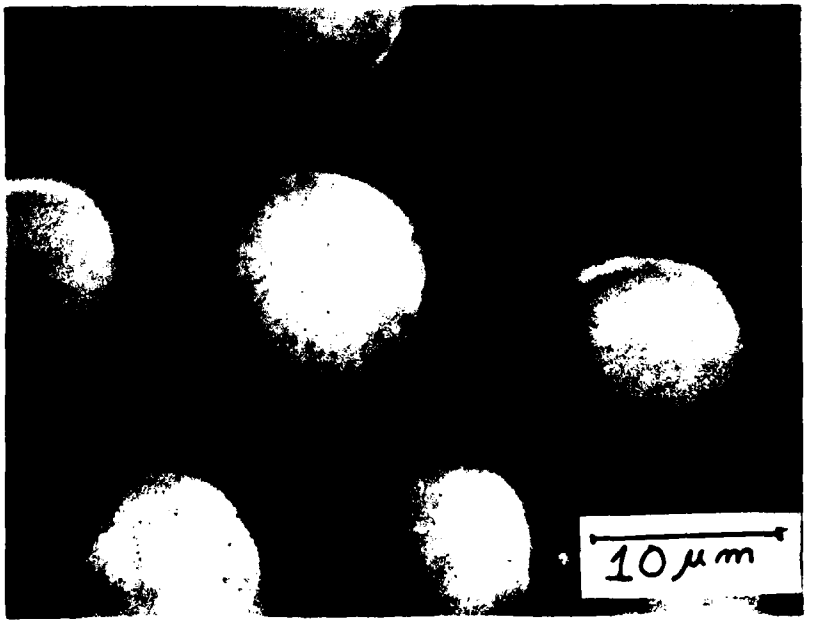


Figure 20. FP/Mg Heated for 100 hours at 350°C.

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AN INVESTIGATION OF INTERFACIAL
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Sung Chor Chin and Marc H. Richman
Brown University
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Final Technical Report AMMRC TR-82-14
March 1982, 32 pp
Contract DAAG46-80-C-0027
D/A Project: 1L263102D071
Final Report 1 Apr 80 to 30 Jun 81

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The interfacial reaction between fibre and matrix materials was studied in G/Al and FP/Mg composite systems. An intermediate layer of reaction product was necessary to prevent debonding in G/Al composites during cooling from the fabrication temperature. The intermediate layer was identified as the spinel $MgAl_2O_4$ in the FP/Mg composite and the effect of this intermediate layer was to provide good bonding and load transfer from matrix to fibre and also to form bridges (necks) cross-linking the fibres. The spinel also acted as a diffusion barrier to reduce the role of fibre degradation by interfacial reaction at service temperatures.

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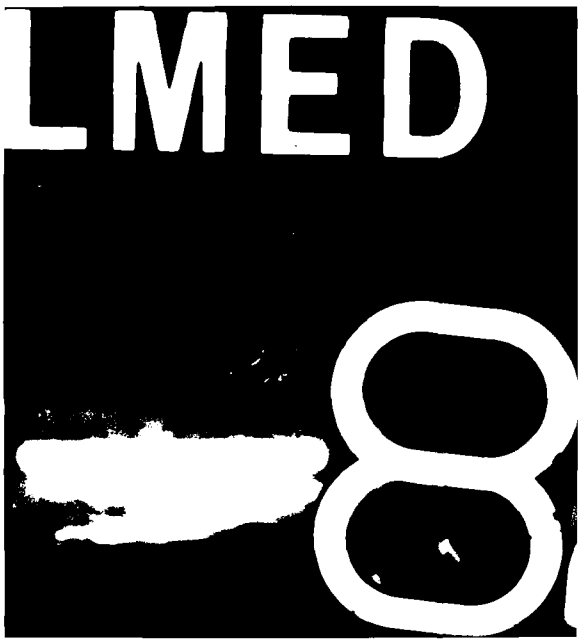
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